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Forensic Exploration of the Mechanical Properties of Basalt Grains in Earthenware

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ABSTRACT

The overall goal of this project is to contribute to reconstruct the innovation mechanisms and development of ceramic production using forensic engineering techniques. Instead of optimizing materials as a driver in modern engineering, here we wish to use these methodologies, but aim to solve questions on advancement in the past fabrication process – and thus ultimately understand the key issues of a less or (un)successful design and subsequent improvement. This paper wishes to address the advantages and constraints regarding to use of basalt in ceramic matrices. By utilizing a standardised set of different test bars comprising different amounts of basalt fired at both 800°C and 1000°C, it can be concluded basalt tempered ceramics have a higher fracture toughness when compared to quartz enriched materials. It is therefore plausible to identify basalt as a good temper material for (ancient) earthenwares in terms of thermal (shock) activities.

Keywords: ancient ceramics, fracture toughness, Levant, basalt temper, experimental archaeology.

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1. Introduction

Material Science in general, and forensic engineering in particular (Carper 2000, Neale 2009), often analyse problems regarding the mechanical failure of a wide set of materials. Frequent questions incorporate the search on how flaws in a material are initiated, but also incorporate questions on stress capability as well as how various variable influence and affect failure stress. As a result of a forensic engineering investigation, understanding the causes of failure can also yield important information to improve the life or performance of different materials.

This forensic approach can be useful in the study of production technology and innovations through time. This paper specifically focuses on tracing the advantages of temper in ceramic materials in order to discover their failing mechanisms opposed to other inclusions and ultimately contribute to reverse engineer broken ceramic objects and the raison d'être of selected impurities. This approach has been mainly been employed in modern ceramic industry and brick manufacturing in order to improve to general properties of the ceramic materials and the use of alternative resources (Stefanov 1991, Ribeiro *et al*. 2002, Zouaoui and Bouaziz 2017)

From the very start of ceramic production, various mineral temper grains were intentionally added by craftsmen to reduce shrinking of the pottery and improve their thermal shock resistance and overall qualities (Bronitsky and Hamer 1983, Biton *et al*. 2014, Kilikoglou *et al*. 1995, Hein *et al*. 2008). Quartz and carbonates are commonly used for this (Allegretta *et al*. 2015), but Levantine communities frequently added basalt to their cooking ceramics (Dornemann 1983), especially extensively from the Iron age II period onwards and might coincide with the practice of new manufacturing procedures and requirements opposed to earlier periods. The main archaeological goal of this paper is to answer the question whether the addition of basalt would enhance the suitability of this pottery and contribute to answer the question of recipe selection by ancient craftsmen. Basalt is by far not a rare resource but is sometimes less accessible in this region (especially the coastal areas) opposed to quartz minerals which are readily and ubiquitous available. Several Levantine craftsmen likely travelled or obtained basalt which has outcrops several days away. Basalt and the presence of this material in (Levantine) pottery has been attested widely and mostly identified as local production, but has only limitedly been questioned on a technological basis (Philip and Baird 2000; Nieuwenhuyse *et al.* 2018). The addition of basalt to cooking ceramics however seems to be preferred over the addition of quartz, although probably more-time-consuming regarding procurement and preparation. The potential beneficial technical aspect of this practice needs however additional research. Many studies deal with the cultural properties of ceramics; the characteristics of technology and provenance of inclusions (Braekmans *et al.* 2017, Ting *et al.* 2018), but few have taken a mechanical angle focusing especially on the effects of the inclusions of basalt (Bronitsky and Hamer 1983, Kilikoglou *et al.* 1995, Müller *et al.* 2015)

This paper aims to change that and study the effect of the addition of basalt on the mechanical and thermal properties of ancient cooking ceramics. To discover why basalt was added to cooking ceramics, several areas where there could be improvements have to be analyzed. For cooking ceramics, one does not want it to break, crack or be damaged prematurely in any way during the firing and cooking processes. There are a range of parameters which should be determined to assess this. In this paper, the most important ones will be discussed, being: fracture toughness, Young's modulus, modulus of resilience and the thermal expansion coefficient. It will be checked if the addition of basalt or quartz will result in a higher fracture toughness than no inclusions (Dlouhý *et al.* 2013). Furthermore, it is predicted that the fracture toughness will be higher at higher percentages of inclusions (Müller *et al*. 2010) and that the thermal expansion coefficient will be in the same range as that of the clay, because this is beneficial regarding limiting the damage caused by thermal shock (NEN 2007), in particular for cooking ceramics. Beforehand, it needs some consideration that clay without inclusions was rarely used in the production of cooking or transport ceramics, but relates mostly to tablewares. The most important reason being the fact that pure clay shrinks much more during the drying and firing processes (Dlouhý *et al*. 2013), and this creates additional stresses in the material and increases the chance of cracking the ceramics.

2. Materials

Outcrops of basaltic rock are a common feature along the Levantine coastal area and the direct inland area of modern-day Israel, Jordan, Palestine and Syria. Outside the Levant basic volcanic composition rocks occur in Egypt, west and east of the Nile Delta. Being a commodity readily available from prehistoric times until the modern period, the use of basalt is apparent in everyday life. Full sized objects (e.g. bowls, statues, royal inscriptions, pestles and mortars) and architectural features are well known but basalt is also commonly attested as temper in ceramic objects (Figure 1).

Figure 1. Microstructure of basalt tempered pottery.

In its current state, the ways in which these basalts were selected, procured and used in the production of ceramics across the Levantine region is the main driver behind this research.

Especially the reason why these basalts were selected opposed to the more readily available quartz and carbonate temper needs further investigations. For testing and determining whether basalt inclusions have an effect on ceramics, a set of testbars are required. Table 1 shows the different sets of test bars that were fabricated using K143 clay by Sibelco (Sibelco 2015). There are 14 different test bars compositions, but as every test is performed three times, 42 test bars are needed for both the flexural and the fracture testing, meaning 84 test bars are required in total.

Table 1. Testbar compositions and amounts.

Grain size		**Mass fraction basalt**		**Mass fraction quartz**
	0%	15%	25%	25%
Fine	800°C	800°C	800°C	800°C
Fine	1000°C	1000°C	1000°C	1000°C
Coarse	-	800°C	800°C	800°C
Coarse	-	1000°C	1000°C	1000°C
Samples with notch	6	12	12	12
Samples without notch	6	12	12	12

The first step is to sieve the basalt and quartz to the desired grain sizes. For the fine grain size, basalt and quartz were filtered to a range of 250-600 μm. For the coarse grain size, a range of 600-1000 μm was taken. For the sorting of grain sizes, a column sieve was used. By weighing the right amount of clay, basalt and quartz, the different compositions of clay were made. Much attention must be given to the mixing of the clay with basalt or quartz. The clay needs to be kneaded thoroughly, so the inclusions are evenly distributed and there are no air pockets inside the clay. Afterwards, the test bars can be given their desired shape. The clay can be inserted into a pre-mode aluminum mould and is then compressed, either by hand or by using a roller. It is important that all test bars are subjected to a similar treatment, to ensure that the test bars will have a consistent density. A subsequent step is to remove the clay outside of the construction and to cut the bars to their desired lengths. All test bars have a height of 1.5 cm, a width of 2.0 cm and a length of 12 cm. These measurements were derived from ASTM C1161-02C, by scaling up the stated dimensions. These dimensions were chosen to ensure that the force until failure was large enough to be adequately determined. Regarding the firing process, the temperature in the oven first rises to 620 °C. A slow rise of the temperature is needed to ensure that all water will evaporate from the test bars and to prevent the creation of cracks, which will influence the test results. After this process, the temperature will rise to either 800 or 1000 °C. Now the test bar is sintered and will need some time to cool down, since fast cooling of the test bars can result in additional cracks.

3. Methods and Parameters

It can be assumed that the main reason to include basalt will be to extend the life expectancy of the cooking ceramics and especially improve heat transmission properties of the vessel (Braekmans and Degryse 2016), which is why only the mechanical and thermal properties will be studied. Specifically, this paper will deal with the effects of different firing temperatures, adding different percentages of basalt with varying grain sizes and thus document their influences. In order to achieve this, this paper will compare the same clay with different inclusions, respectively 0%, 15% and 25% basalt and 25% quartz, different firing temperatures, 800 ˚C and 1000 ˚C, and different grain sizes, ranging from 250 µm to 600 µm and 600 µm to 1000 µm, in order to discover the significance of each aspect. The 0% and 25% quartz tempers serve as the control groups so that the absolute effect of adding basalt can be studied and basalt can be compared with quartz, the most commonly used temper. For all the tests done, test bars with dimensions of 120 x 20 x 15 mm were used. For each different ceramic composition, three test bars were made and tested to ensure no accidental faults would lead to false conclusions. In the end this means a total of 84 test bars were tested for this study.

3.1 Fracture Toughness Measurements

One of the most important material properties which should be considered in order to help prevent failure of cooking ceramics, is the fracture toughness. The fracture toughness is important when working with ceramics, as there will always be minuscule cracks present in the material. The fracture toughness will determine if those pre-existing cracks will propagate into a larger crack. To determine the fracture toughness, a three-point flexural test is used with the ASTM 1820 (ASTM 2009) and ASTM C 1421 (Salem *et al.* 2005) standards as global guidelines. By giving the test bars a single edge notch of 2 mm depth and 0.5 mm width, the bars were given an artificial pre-existing crack, which was deeper than any (micro)cracks that might already present. Afterwards, the test bars were subjected to a tensile load until a maximum force, at which the crack propagated, and the test bars subsequently failed. The span of the clamped test bars was set on four times the thickness of the bars, resulting in a span of 6 cm. With the measured height, width, and notch depth the fracture toughness can be determined, using formula (Bower 2012) (1).

$$K_{IC} = \frac{4P_f}{w}\sqrt{\frac{\pi}{h}}[1.6(\frac{a}{h})^{\frac{1}{2}} - 2.6(\frac{a}{h})^{\frac{3}{2}}$$

$$+12.3(\frac{a}{h})^{\frac{5}{2}} - 21.2(\frac{a}{h})^{\frac{7}{2}} + 21.8(\frac{a}{h})^{\frac{9}{2}}] \quad (1)$$

For setting up the test machine, a cross-head displacement rate of 0.1 mm/min (Faber *et al.*

1981) was used. To make sure the cross-head actually came into contact with the test bar, a preload of 10 N was applied.

3.2 Three-Point Flexural Test

Another property that influences if the cooking ware will fail is the Young's modulus. This property determines the elastic behavior which a material will show upon an applied load. For cooking ware it is important that it will not break at the slightest form of impact, but that the energy will be absorbed. To determine the Young's modulus a three-point flexural test without notch was used, with the EN 843-2 (ASTM 2009) standard as a global guideline. Again the span was set on four times the thickness of the bars, resulting in a span of 6 cm. By measuring the maximum force and deflection at breaking, the Young's modulus can be determined. This can be done by means of formula (NEN 2007) (2), in which the span length and the test bar width and height are also needed.

$$E = \frac{F_m L^3}{4wh^3\delta} \qquad (2)$$

For setting up the test machine a cross-head displacement rate of 0.1 mm/min (NEN 2007, Faber *et al*. 1981) and a preload of 2 N were used.

One more material property that will be calculated is the modulus of resilience (Ashby 2005). The modulus of resilience gives the amount of energy the material can absorb before it deforms plastically. The modulus of resilience can be calculated using formula (3) and is given as U_r, in units of $J{\cdot}m^{-3} \cdot 10^4$. Furthermore, σ_y is the maximum stress at which the material still deforms elastically.

$$U_r = \frac{\sigma_y^2}{2E} \quad (3)$$

Since the dimensions of the produced test bars differed minimally with each test bar, solely comparing the force required to break it is not sufficient. However, when comparing stresses, these different dimensions are taken into account, resulting in fair and comparable results. To improve the validity of the results of the test bars, three test bars were tested with each test. The deviation of the corresponding results is examined using statistical software, such as MATLAB and R, to check for normality of the results and to support the drawn conclusions. For both bending tests, with and without a notch, sets of three test bars were used. The mean and the standard deviation of the corresponding maximum stresses were calculated by using MATLAB. By taking the ratio of the standard deviation to the mean, the relative standard deviation could be obtained. A histogram of these standard deviations, combined with the curve of a normal distribution with the same mean and standard deviation can be seen in Figure 2.

The statistical software R was used to test for normality using several methods. If the calculated p-values of these tests are higher than 0.05, which is the a-level, the null hypothesis that the sample data are normally distributed, is not rejected. This means that the results could be seen as approximately normally distributed. The Jarque-Bera test resulted in a p-value of 0.8944 and the Shapiro-Wilk test produced a p-value 0f 0.9006, which corresponds to the assumption of an approximate normal distribution. This conclusion is also supported by the small deviations from the red line in the quantile-quantile plot, which is a graphical test for normality, as can be seen in Figure 3. This justifies the plot of the normal distribution in combination with the histogram in Figure 2.

Figure 2. Distribution of the relative standard deviation.

Figure 3. Normal quantile-quantile plot.

4. Results and Discussion

The addition of inclusions in a material will influence the way the material behaves at higher temperatures. The thermal expansion coefficient of the inclusions will have to be in the same range as that of the base material. If not, the difference will create internal stresses and weaken the material instead of strengthening it. This phenomenon is known as thermal shock (Faber *et al*. 1981). For the ceramic material used in ancient cooking wares it is even more important because of its brittleness. Due to its composition and fabrication process, terracotta (CES Edupack 2016) can be used as a reference for the clean ceramic material. The thermal properties of basalt (Dane 1942) and quartz (Skinner 1966), another widely used material for inclusions, can be found in previous research projects (Huotari and Kukkonen 2004).

The linear thermal expansion coefficient is defined as α and is given in m/(m·K) $\cdot\ 10^{-6}$. Terracotta has a linear thermal expansion coefficient of 4-10, basalt 5.4 +/- 1 and quartz 16.7. The coefficient of basalt thus lies within the lower range of that of terracotta, whereas quartz exceeds the upper range. Therefore, it can be concluded that the addition of basalt should theoretically result in less thermal shock than the addition of quartz.

As can be observed in Figures 4-6, ceramics fired at 1000 °C with no inclusions have the highest fracture toughness, modulus of resilience and Young's modulus. Moreover, ceramics fired at 1000 °C perform multiple times better than ceramics fired at 800 °C. Furthermore, the addition of basalt leads to a significantly higher fracture toughness and modulus of resilience than the addition of quartz, although there are no large differences in the Young's modulus between basalt and quartz inclusions. Moreover, a mixture with 15% basalt leads to a larger modulus of resilience than a mixture with 25% basalt. Additionally, a small rise in fracture toughness is observed when using the coarse grain size. The same can be observed at 800 °C, although on a smaller scale. It is also interesting to mention that the ceramics with coarse inclusions had some residual stresses present, meaning that upon failure, the test bar did not straight away experience total failure, but could still hold a load, although it was significantly lower than the maximum load.

Figure 4. The fracture toughness of the different test bars. The x-axis labels the compositions of the test bars, whereas the y-axis indicates the fracture toughness in MPa•m0.5. Circles only indicate a set of three.

Figure 5. The Young's modulus of the different test bars. The x-axis labels the different compositions of the test bars, whereas the y-axis indicates the Young's modulus in GPa. Circles only indicate a set of three.

Figure 6. The modulus of resilience for the test bars without notches. The x-axis gives the different compositions of the test bars, whereas the y-axis indicates the modulus of resilience in J • m−3 • 104. Circles only indicate a set of three.

Adding inclusions, such as quartz, further reduces the Young's modulus of the ceramics, which concurs with the existing reference data (Kilikoglou *et al*.1998). Furthermore, higher firing temperatures lead to improved material properties, in agreement with the existing literature (Kilikoglou *et al*. 1995). It was also seen that test bars without temper were affected most by shrinking during the firing process, since temper reduces the shrinking significantly (Biton *et al*. 2014).

It should be noted however, that during production of the test bars, the human factor likely played an important role as in the past. Although the desired dimensions of the clay bars before firing were clear and there was continuous monitoring and effort to produce them as accurately as possible, the dimensions differed slightly when shaping them. More specifically, the force and precision used when fabricating the clay bars might differ, thus leading to slightly different densities or inhomogeneities in the clay. In combination with the firing process in the kiln, where shrinkage occurred, this resulted in variable dimensions. Although the different dimensions were accounted for in the results section by looking at the stress, inhomogeneities will always remain in this type of material, and therefore cause some degree of inaccuracy.

The results for the Young's modulus differ significantly from the reference values for terracotta found in the literature (CES Edupack 2016). Moreover, direct comparison of the results is difficult, since the clay used for the test bars differed from the clay used in other research. Nevertheless, difference can be compared on a relative scale. Errors produced by the machine or made in the calculations are highly unlikely, since the equipment was accurately calibrated, and the calculations were thoroughly checked. The force-displacement curves often show initial non-linear behavior. Since the Young's modulus is based on the assumption of linear behavior, this introduces an error. Most likely, this non-linearity is caused by the relatively large size of the test bars, since more accurate results seem to have been obtained by using smaller test bars (NEN 2007). Furthermore, the inhomogeneities, introduced during the production process of the test bars, played a significant role in determining the Young's modulus, since less force might have been required to accomplish a certain displacement, therefore resulting in a lower Young's modulus.

There is not a lot of data on the fracture toughness, but it can be seen that the data in this study agrees with the data found in the literature (for instance, Müller *et al*. 2014). Furthermore, the Young's modulus seems to be influenced mainly also by clay type of the base material as gathered from (un)tempered results of K143, and published phyllite and granite rich clay samples (Müller *et al*. 2014) that differ something significantly. Overall, the data from this study and base clay yielded a smaller Young's modulus in comparison to the Young's modulus of other studies.

5. Conclusion

As a result of the research done in this paper, it can be concluded that the addition of basalt is preferable in multiple ways. Addition of basalt results in a higher fracture toughness, which is a very important feature for the life expectancy of porous ceramics. Furthermore, the addition of basalt prior to firing results in less thermal shock compared to quartz, due to its more favorable thermal expansion coefficient. Finally, ceramics with basalt inclusions can absorb more energy before failure, according to the calculated values for the modulus of resilience. This gives a clear indication of why ancient craftsmen made the choice and effort to add basalt, even though it was not always readily available in several areas (especially coastal) in the immediate vicinity of the sites and required more energy to procure and process.

Basalt thus yields a significant improvement to commonly used quartz grains, especially regarding the fracture toughness. However, to assess variability it is necessary to note that the process of fabricating the test bars is can be prone to human errors. It is therefore recommended to determine the protocols for the fabrication of the test bars. The purpose of this protocol is to standardise the process and ensure that each test bar is subjected to the same conditions, meaning that each test bar fabricated is as consistent as possible. Another improvement regarding the accuracy is to use a four-point flexural test, instead of a three-point flexural test as utilised here. With the four-point flexural test it is possible to more accurately determine the Young's modulus, because stresses will be uniform in the center of the material. Moreover, this initial research was mostly focused on mechanical properties, it is advised to also examine other areas in which the addition of basalt could influence material properties, such as thermal conductivity.

The most likely cause of the addition of this material is due to mainly the improvement in thermal expansion coefficients of basalt relative to that of the ceramic itself, which would be beneficial both for both resistance to thermal shock and heat transmission. It is also advised to incorporate a further detailed analysis of the microstructure of the ceramics near the inclusions to determine the type of failure propagation of the cracks.

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